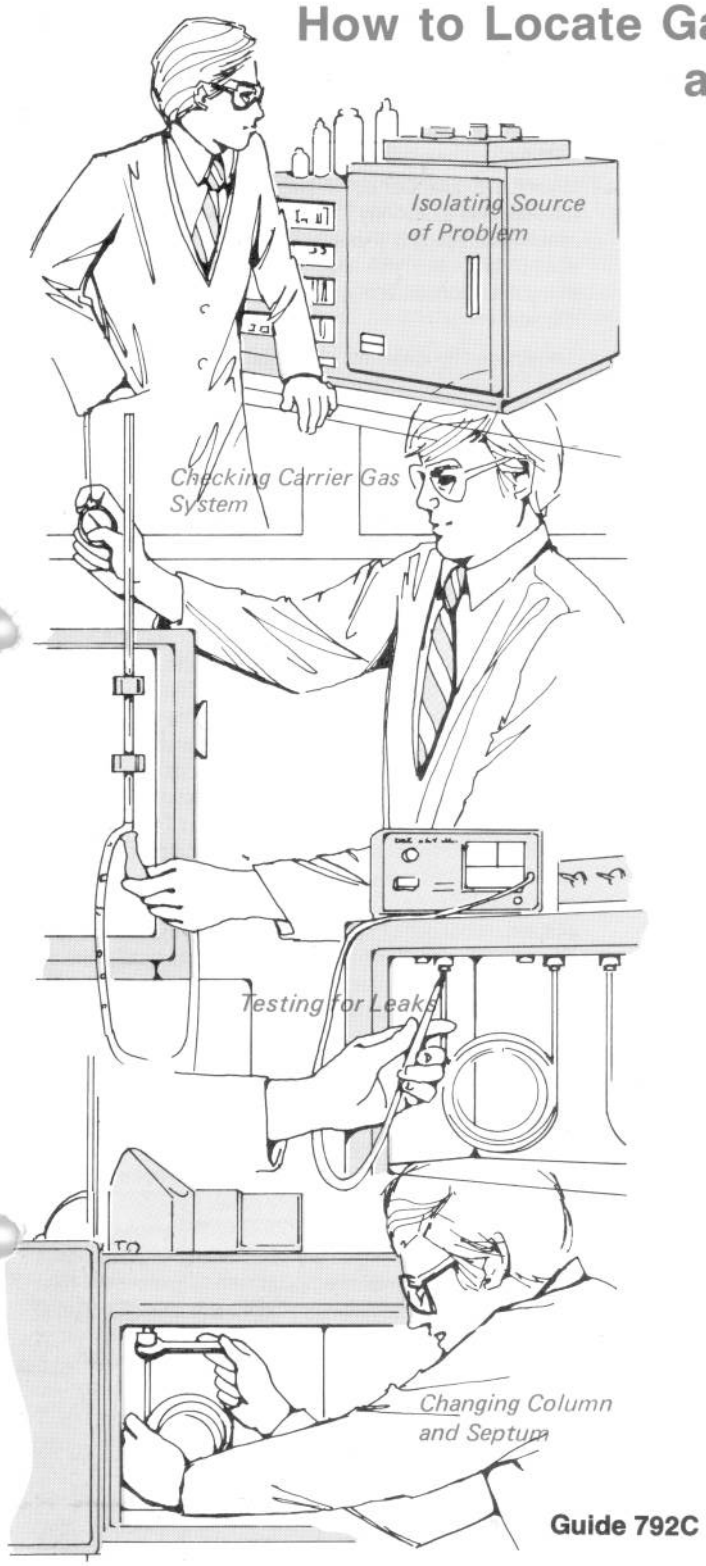
TROUBLESHOOTING GUIDE

How to Locate Gas Chromatography Problems and Solve Them Yourself . . . .



Guide 792C

By applying a systematic approach to troubleshooting, you can solve many of your own GC problems. The real task is identifying the cause of a problem in the shortest possible time. This guide outlines techniques that will enable you to troubleshoot your gas chromatography step-by-step. You'll reduce repair costs and instrument downtime.

Suggestions for Effective Troubleshooting

Certain basic procedures make troubleshooting faster and easier. Most important are close observation of operating parameters and a good record keeping system (temperatures, flow rates, chart speeds, column type, stationary phase type and amount, solid support type and mesh size, etc.). Also of primary importance is a reference standard containing known concentrations of the components in your sample. The reference standard should contain no extraneous, unknown components. Many hours can be wasted hunting problems within an instrument or column, when the problem is in fact the sample being analyzed. If your chromatographic system separates the reference standard well and reproducibly, any existing problem is most likely related to the sample.

Your troubleshooting will progress more smoothly if you have on hand:

1. *A duplicate analytical column, one you know will provide acceptable separation under proper conditions* — Try this duplicate column in your malfunctioning system. If it corrects your problem, the problem is related to the original column.
2. *A new syringe to help isolate the source of ghost peaks* — Repeat the analysis with a new, clean syringe. If your trouble disappears, the problem has been isolated to a defective or dirty syringe used during the original analysis.
3. *Snoop® or other leak detection aids* —Use these to ensure that your entire system is free of leaks, mandatory for proper operation. (NOTE: Don't use these liquids with capillary columns.)
4. *Spare septa and high temperature septa* — These help to identify problems with reproducibility or ghosts caused by leaking or bleeding septa. Simply replace your septum with a new duplicate septum or with a higher temperature septum. If the symptom disappears, the trouble was a leaking or bleeding septum.
5. *Detector cleaner* — A dirty detector creates noisy baselines. Flame ionization detectors (FIDs) may be cleaned using either Freon® TF, an in-place cleaner, or an ultrasonic bath filled with an immersion cleaner.
6. *Thermometer* — To verify the oven temperature, ruling out a defective temperature control.
7. *Spare ferrules* — Often necessary to eliminate leaks in connections.
8. *Soap bubble meter* — To check your gas flow.
9. *Spare recorder and electrometer cables* — To eliminate the recording system as a source of trouble.
10. *Instrument manual.*

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SUPELCO, INC. • Supelco Park • Bellefonte, PA 16
Phone (814) 359-3441 and (814) 359-344
TWX 510 670-3600 • FAX 814-359-3044

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ISOLATING THE PROBLEM SOURCE

There are five major sources of problems in gas chromatography: (1) the operator, (2) the sample, (3) the column, (4) the electronics, and (5) the gas flow system. Various problems can stem from any one of these sources. Eliminate them one-by-one to isolate the actual cause of the problem.

To define your problem, first refer to the Troubleshooting Table on page 5. Using the symptoms index at the front of the table, locate your trouble symptom. If there is more than one symptom, note the possible causes for each. If one cause is common to all symptoms, this is most likely the source of your problem. (Note that while the troubleshooting table contains most of the symptoms you will encounter, it cannot cover all potential problems.) When you cannot find a rapid solution using the troubleshooting table, you must systematically isolate the trouble:

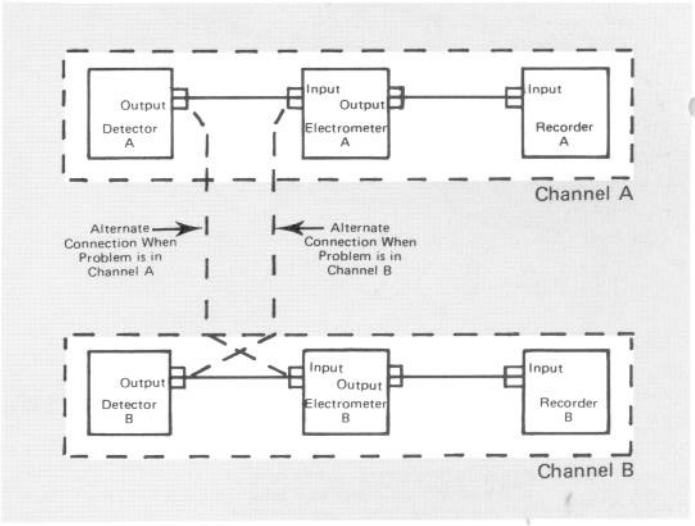
1. Carefully note the symptoms (e.g. broad peaks, unresolved peaks, long retention times).
2. Rule out operator error by double checking all operating parameters, such as temperature, carrier gas flow, column description, etc.
3. Check for a sample problem by injecting a reference standard. If you get a good chromatogram, the problem is most likely sample related. If not, the problem is probably column or instrument related.
4. Check for a column problem by replacing the column with a duplicate column, one known to provide good results under proper conditions. If good results are obtained, the problem is related to the original column. If the symptom persists, the problem is related to the instrument.
5. Isolate equipment related problems by listing the equipment systems which can cause the observed symptoms (e.g. for broad peaks with long retention times: (1) carrier gas system, (2) column heating system, and (3) injection port heating system). Next, isolate the problem within the suspected systems.
6. Isolate possible electronic system malfunctions (detector, electrometer, recorder, and associated wiring) by performing the following checks:

   NOTE: If your instrument is equipped with dual channels (detector, electrometer, recorder, etc.), see paragraph (c).

   (a.) Check the recorder by setting the gas chromatograph's attenuation to infinity. The recorder pen should go to electronic zero. If the symptom (baseline drift, noise, etc.) disappears, the recorder is not the problem. If the symptom continues, refer to the recorder instruction manual.

   (b.) To isolate the detector (FID) as the source of trouble, turn off the instrument and disconnect the cable (at the detector end) from the detector to the electrometer. (Note: To prevent inducing extraneous noise onto the cable, it may be necessary to install a *coaxial* cap on the free end.) If the symptom disappears when power is on, the problem is the detector. If the symptom continues, disconnect the same cable at the electrometer. If the symptom *now* disappears, the cable is defective. Replace it. If the symptom still continues, refer to the electrometer instrument manual.

   (c.) If your chromatograph is equipped with dual detector channels, you have a simple but effective alternate means of identifying the problem source using the second, or parallel, detector and electrometer. If the symptom occurs in channel A, disconnect at the detectors (shown below) the cables which connect channels A and B detector outputs to channels A and B electrometer inputs. Reconnect the cable from electrometer B input to the output of detector A. This applies the signal from the detector output of channel A to the electrometer input and recorder of channel B. If the symptom does not appear on recorder channel B after this cable change, then either the electrometer, recorder, or cables of channel A are defective. If the symptom is not eliminated, the detector of channel A is the problem source.



7. Check the carrier gas system for possible problems. Refer to the following section, Checking the Carrier Gas System.

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CHECKING THE CARRIER GAS SYSTEM

A typical carrier gas system is illustrated at the bottom of this page. The most common problem within this system is insufficient carrier gas flow through the chromatographic column. This is generally caused by (1) insufficient source pressure, (2) leaks, and/or (3) an unusually large pressure drop across one of the components in the system.

Verify the column carrier gas flow at the detector exit using a soap bubble flow meter and a stopwatch. (We do not recommend using rotameters for measuring gas flow because specific rotameters are required for each type of gas used, and they possess a definite linear response range with pressure changes.)

To verify that the carrier gas flow rate is properly adjusted, the time required for the soap bubble to travel a specified distance at the desired flow rate should be calculated as follows:

$$\text{Time (sec.)} = \frac{\text{Volume Observed (cc)} \times 60 \text{ sec./min.}}{\text{Desired Flow (cc/min.)}}$$

Where:

Time (Sec.) = time required for the bubble to travel the observed distance.

Volume Observed = volume indicated by the soap bubble flow meter.

Desired Flow = the rate specified by the method being used.

Example: Obtain a flow rate of 20 cc/min. using a 10cc soap bubble meter.

$$\text{Time (sec.)} = \frac{10\text{cc} \times 60 \text{ sec./min.}}{20\text{cc/min.}}$$

$$\text{Time (sec.)} = 30 \text{ sec.}$$

If the time required is not equal to the calculated time, adjust the carrier gas flow rate. If sufficient flow cannot be obtained by adjusting the flow control valve, the problem is probably due to inadequate source pressure (observed at P1). Increasing this pressure will usually provide adequate flow. Normally, a source pressure of 60psig is sufficient for 6 to 12 foot columns. Remember, increasing the column length, oven temperature, and/or flow rate will require raising the source pressure. The source pressure is particularly important if you are using a temperature program, since the pressure must be 10-15psig in excess of the column pressure drop at the maximum temperature. This pressure difference allows the differential flow controller to function properly. If the correct pressure difference is not maintained, carrier gas flow will drop drastically at elevated temperatures.

Other common causes of inadequate gas flow are leaks in the system and large pressure drops across one or more of the system components. The use of pressure gauges can save considerable time when isolating these problems. Some common leak points are: column connections, the septum, and connections for the various valves and gas purifiers.

A pressure gauge installed between the particle trap and the injection port (P3 in the gas system illustration) indicates column head pressure. A low reading at this point indicates a leak between P3 and the detector outlet (e.g. a defective column, septum, etc.) or a large pressure drop across a previous component (e.g. a plugged oxygen trap). A high reading at this point indicates an over-tightened septum, dirty detector, too-tightly packed column, etc. Similarly, a pressure gauge at P2 will reveal an exhausted oxygen trap (low pressure readings indicate a larger than normal pressure drop across the trap). Routine observation of this pressure enables you to determine when the carrier gas purifier should be changed.

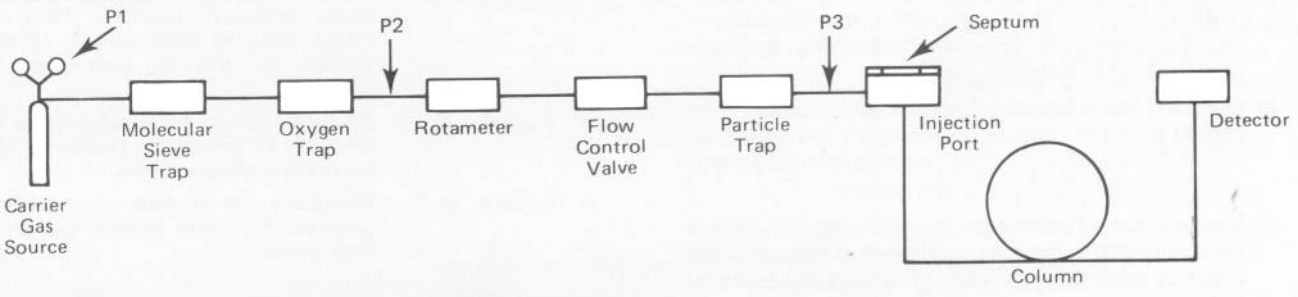
NOTE: Many chromatographs have crimps in the carrier gas line between the flow controller and the injection port or employ capillary tubing with a small internal diameter. Consequently, the pressure reading at point P3 will be different from the column head pressure reading taken through the septum. These restrictions can also make it difficult to obtain sufficient carrier gas flow, particularly when converting instruments for use with capillary columns.

TESTING FOR LEAKS

The most common method of leak testing is to apply liquids such as Snoop or HT-Leak Detector, and then watch for bubbles to appear. Exercise care when using these liquids since they can contaminate your chromatographic system, causing unstable baselines and "ghost peaks" in subsequent chromatograms. (These liquids can enter the GC system through an aspirator effect.)

Several devices are available for leak testing, such as thermal conductivity leak detectors and pressure gauges. (The latter were discussed in the previous section.) When pressure guage readings indicate a possible leak, take the appropriate corrective action (such as tightening the fittings which would cause this abnormal pressure reading) rather than verifying the leak point with Snoop or HT-Leak Detector and possibly contaminating the chromatographic system. A specific technique for testing small septum leaks while avoiding contamination employs the Supelco leak tester, a plastic capillary tube with two conical ends. One end is dipped into Snoop, and capillary action pulls a small amount of Snoop into the tube. If a leak is present, bubbles appear at this end when the opposite end is pressed against the septum nut.

Typical Carrier Gas System



PROBLEMS RELATED TO COLUMN & SEPTUM REMOVAL & INSTALLATION

When changing columns or septa, it is important that you first turn off the chromatograph oven and allow the column to cool for 10 to 15 minutes, then turn off the carrier gas. Cooling the column and turning off the carrier gas protects your column in two ways: allowing the column to cool prevents oxidation of the column packing which can occur when a hot column is exposed to oxygen in the air. Allowing the column pressure to drop to ambient pressure prevents the packing from popping or blowing out of the column ends. A sudden change in pressure, when a column or a septum is removed with the carrier gas flowing, can blow packing from the column.

NOTE: When storing columns, cap the ends with metal Swagelok® caps to prevent diffusion of air (and subsequent oxidation) into the column. Plastic caps *do not* prevent diffusion of air into the column.

Improperly installed columns and septa are a frequent source of leaks, and are the most common cause of glass column breakage. Incorrectly tightened septum nuts present problems such as excessive septum bleed, premature occurrence of septum leaks, and low carrier gas flow rates.

Supelco offers two types of torque wrenches to help ensure correct installation of columns and septa. The Glasrench, used for installing columns, is available in two different torque settings to provide the correct torque for the various types of ferrules. This saves both analysis time and money by eliminating leaks, over-tightening, and column breakage. Select and use the appropriate Glasrench for the ferrule you are using.

The Supelco septum nut torque wrench ensures that the correct torque is consistently applied when installing septum nuts.

SAMPLE INJECTION

Improper sample injection can cause many problems in gas chromatography. To ensure that your injections are accurate and reproducible, the following general guidelines and procedures are recommended:

A. **Syringe Size.** Always use a syringe large enough that the desired sample volume does not fill it to capacity, and small enough that the sample volume is not less than approximately 10% of its capacity.

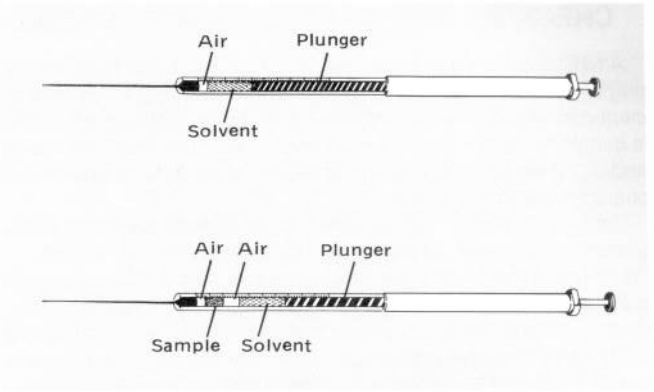
B. **Injection Technique.** Sample injection should be smooth and rapid with quick removal of the syringe after injection, in order to avoid peak broadening.

C. **Sample Size Reproducibility.** Many problems in chromatography result from difficulties in reproducing the size of a sample. Some techniques which will help ensure reproducible samples are discussed below:

1) **Automatic Injectors.** These devices improve sample reproducibility by virtue of consistent mechanical operation. Each step (syringe cleaning, sampling, and sample injection) is repeated precisely.

2) **Sampling Valve Injection.** Sample reproducibility is improved because chances for variability are greatly reduced. Sample size is determined solely by sample loop size, and injection is rapid and precise.

3) **Solvent Flush Technique.** The following technique is recommended to reduce the problem of irreproducible injection volumes when making syringe injections by hand:



(a) Eliminate sample hang up in the needle by first cleaning the syringe, then drawing in a small aliquot of solvent.

(b) Remove the syringe from the solvent and draw in a small amount of air.

(c) Draw in the desired amount of sample.

(d) Remove the syringe from the sample and draw in a little more air.

(e) Verify the amount of sample in the syringe barrel. This is only possible with syringes that do not have plungers in the needle.

(f) Quickly and smoothly inject sample into the chromatograph.

4) **Syringes With Needle Plungers.** Improve sample reproducibility by using a syringe with a plunger in the needle. This eliminates sample retention in the needle dead volume. The solvent flush technique, (3) above, may be useful, since a small amount of sample hang-up can still occur.

OTHER USEFUL PUBLICATIONS

In addition to information presented in this guide, more helpful tips are available to save time and money in chromatography. Some of this information is provided FREE, upon request, in the following Supelco technical bulletins:

- **Bulletin 739** — Reviews problems caused by using impure carrier gas, and methods of extending column life.
- **Bulletin 780** — Discusses the selection of a septum which will resist leaks and exhibit low bleed at high operating temperatures. This bulletin provides test results demonstrating the quality of Thermogreen™ LB-1 septa.
- **Bulletin 783** — Provides information pertaining to dirty flame ionization detectors (FIDs), and offers hints to help prevent contamination, as well as instructions for cleaning.
- **Bulletin 741** — Reviews various types of ferrules and methods of preventing problems when connecting glass columns.
- **Bulletin 814** — Discusses use of wide bore capillary columns in packed column gas chromatographs.

ABBREVIATIONS

ECD – Electron Capture Detector
FID – Flame Ionization Detector
FPD – Flame Photometric Detector
GC – Gas Chromatography
NPD – Nitrogen Phosphorous Detector
TCD – Thermal Conductivity Detector

SYMPTOMS INDEX

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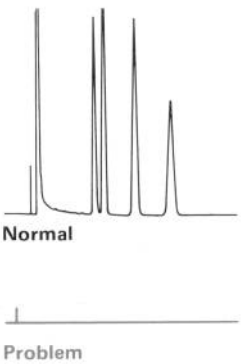
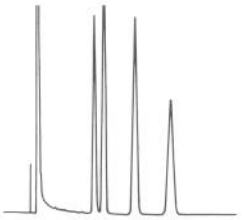
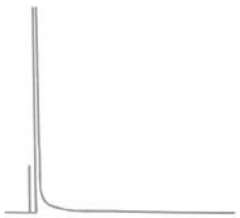
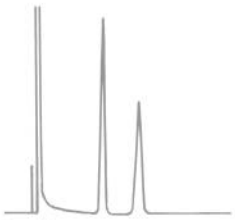
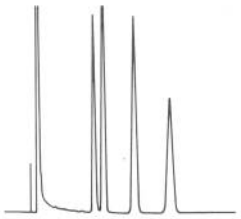
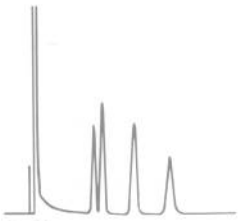
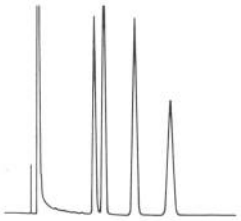
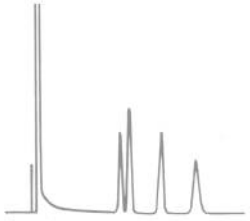
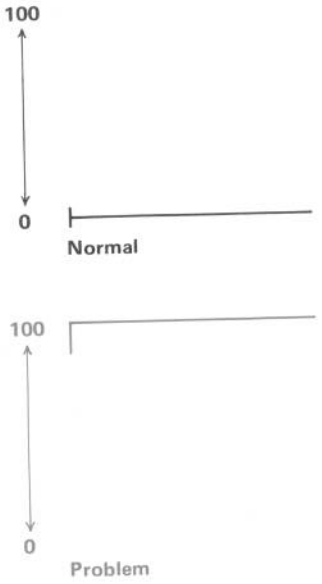
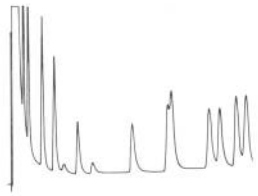
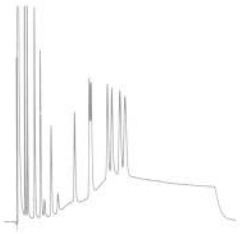
TROUBLESHOOTING TABLE

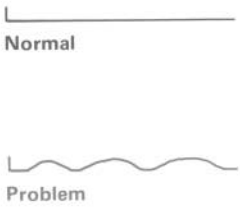
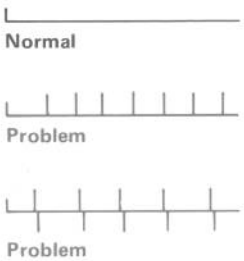
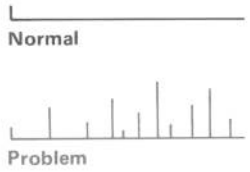
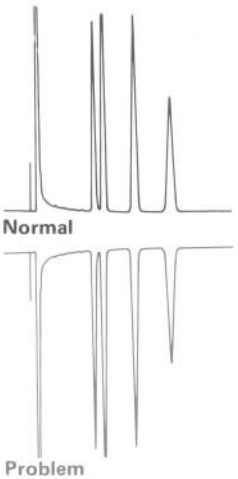
Symptom	Possible Cause	Remedy
Symptom No. 1		
No Peaks 	1. Detector or electrometer power off or fuse blown	1. Check detector, electrometer setting, and fuses.
	2. Sample injected in wrong column in multiple-column chromatograph	2. Re-inject sample in proper column.
	3. Flame ionization detector (FID) not lit	3. Check FID with mirror over exhaust. If lit: H_2O condensates on mirror. If not lit, light flame. Check H_2 and air flows.
	4. No carrier gas flow	4. Measure carrier gas at detector or column exit. If no carrier gas flow is present, check for leaks or obstructions at column connection and septum. Check for adequate pressure at tank and ensure septum is not over-tightened (flow cut off by septum obstructing the column).
	5. Defective syringe	5. Replace syringe

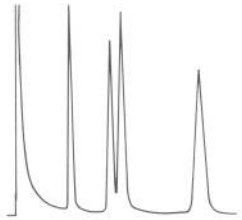
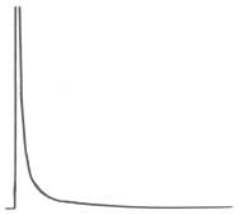
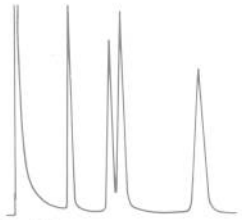
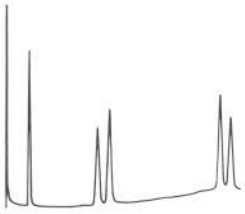
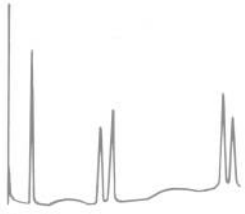
Symptom	Possible Cause	Remedy
(Symptom No. 1, cont'd.)	6. Column or septum leak	6. Replace the septum. Check column connections.
	7. Injection port temperature too low, sample not vaporized when analyzing high boilers	7. Increase injection port temperature, (but not in excess of liquid phase temperature limit), or inject sample directly onto column packing.
	8. Defective recorder	8. Check recorder connections. Check recorder zero. Troubleshoot recorder according to recorder manual.
	9. Defective detector, electrometer, or cables	9. Check collector voltage and connections per instrument manual.
	10. Bad connection between FID collector and voltage source	10. Check "collector spring clip" connection.
Symptom No. 2		
Solvent peak only, or missing peak  Normal  Problem  Problem	1. Sample too dilute	1. Check system by injecting with known standard. If okay, increase sensitivity or inject larger or more concentrated sample.
	2. Column or septum leak	2. Check for leaks (see page 4). Tighten connections. Replace septum.
	3. Incorrect temperatures (a.) Injection port or column temperature too low (sample not vaporized)	3. (a.) Ensure that the selected column temperature setting is correct for column being used and the sample being analyzed. Then verify that the oven is operating at the selected temperature. Increase the temperature as required.
	(b.) Injection port temperature too high for thermally labile compounds	(b.) Decrease injection port temperature.
	(c.) Column temperature too high, sample eluting in solvent peak	(c.) Decrease column temperature.
	4. Flow rate incorrect	4. Measure column flow rate and adjust if necessary (see page 3).
	5. Sample adsorption by column or glass wool	5. Inject standard on known "good" column. If okay, original column is bad. Use properly treated glass wool (i.e., H_3PO_4 for free acid analysis, silicone-treated for other compounds). If sample has never been analyzed and is active chemically, you may need a special column.
	6. Column cannot separate components from solvent	6. Change column or solvent.

Symptom	Possible Cause	Remedy
Symptom No. 3		
Low response (all) – correct retention time  Normal  Problem	1. Poor injection technique 2. Wrong sensitivity setting or sample size too small 3. Defective syringe 4. Septum leak 5. Injection port temperature too low for sample 6. FID only – H_2 or air flow incorrect 7. FID only – low O_2 level in compressed air 8. FID only – faulty connection between FID collector and voltage source 9. Dirty electron capture detector (ECD) 10. (a.) Thermal conductivity detector (TCD) carrier gas flow rate is incorrect (b.) Cell voltage is incorrect 11. Adsorption of sample by column, glass wool, tubing, etc. 12. Flame photometric detector (FPD) only – hydrocarbon eluting with sample, causing diminished response due to quenching effect	1. Use correct syringe size and solvent flush techniques (see pages 4 and 5). 2. Check and correct if necessary. Inject standard sample for comparison. 3. Use new syringe. 4. Replace septum. 5. Increase injection port temperature. 6. Measure and correct if necessary. 7. Replace air tank. 8. Clean "collector spring clip" with emory paper. 9. Clean per instrument manual. 10. (a.) Measure and adjust carrier gas flow if necessary. (b.) Refer to instrument manual. 11. Use deactivated column materials. 12. Check with known standard (hydrocarbon free) and change to column that will separate hydrocarbons from components of interest.
Symptom No. 4		
Low response – long retention time  Normal  Problem	1. Low carrier gas flow rate 2. Carrier gas leak at septum or column connections 3. Column temperature too low 4. Column worn out or conditioned at too high a temperature	1. Measure and adjust carrier gas flow rate if necessary (see page 3). 2. Check for leaks and correct if necessary (see page 4). 3. Increase column temperature. 4. Verify column temperature and stationary phase temperature limits. Check sample with known good column. Repack first 6" of column or replace column.
Symptom No. 5		
Quantitation not reproducible a. Retention time okay. Components with longest retention time show low value when using "normalization" techniques.	1. Sample incorrect a. 1. Incomplete sample injection 2. Injection port or column temperature too low 3. Incorrect slope sensitivity with electronic integrator	1. Verify using known standard. a. 1. Use solvent flush techniques. 2. Increase temperature (see page 4). 3. Adjust slope sensitivity.

Symptom	Possible Cause	Remedy
(Symptom No. 5, cont'd.)		
b. Retention time okay. Different components not yielding similar peak areas for same amount	b. 1. Differing detector response for different components	b. 1. Determine correction factors and/or use internal standards techniques.
	2. Adsorption of components by packing, glass wool, tubing, or transfer lines	2. Use deactivated system.
c. Quantitation varies for one component eluting over wide time span, even using internal standard technique	c. 1. Internal standard not compensating for all components in sample	c. 1. Use multiple internal standards.
	2. Slope sensitivity of integration not high enough for late eluters	2. Use multiple internal standards.
d. Inconsistent quantitation for same sample on successive analyses	d. Insufficient resolution of peaks, or peak tailing	d. Modify operating parameters or replace column to improve resolution and eliminate tailing.
e. Low values for minor compounds	e. Sample size too small for accurate counting by integrator	e. Increase sample size or electrometer range setting.
f. Increased peak response with successive injections	f. Adsorption of components and saturation of active sites with sample (priming the column)	f. Use deactivated system.
Symptom No. 6		
Baseline off-scale, cannot zero  	1. Column not conditioned properly, contaminated column, or temperature too high	1. Verify by reducing column temperature to ambient. If baseline is normal, check system with good column. If okay, recondition "bad" column.
	2. Recorder problem	2. Set attenuation to infinity. If recorder goes to electrical zero, it's okay. If it does not go to zero, troubleshoot recorder per recorder manual.
	3. Septum leak	3. Check septum for leaks and correct leaks if required (see page 4).
	4. Wrong gases (e.g., argon/methane) with flame	4. Verify gases are correct for instrument and detector as specified in manual.
	5. Contamination	5. Turn off injection port heat. If zeroing capability returns, clean injection port liners, etc.
	6. Too much or too little gas flow	6. Check and adjust to within manual specifications.
	7. Imbalance in column flow if using TCD	7. Check and adjust as required.
	8. Contaminated detector, such as nitrogen phosphorous detector (NPD) with Snoop, or ECD with chlorinated solvents	8. Avoid sources of contamination. (i.e. Don't use Snoop with NPD or chlorinated solvents with ECD.)
	9. Electrometer or detector problem	9. Troubleshoot instrument per manual.

Symptom	Possible Cause	Remedy
Symptom No. 7		
Baseline drift  Normal  Problem	1. Carrier gas flow changing with temperature during temperature programming 2. Septum or column leaks 3. Septum bleed or septum fragments in column 4. Column bleed or contamination 5. Gas flows not within minimum/maximum limits (including H_2 and air on FID) or poorly regulated flow 6. Insufficient instrument warm up time or temperature equilibration time 7. Defective electrometer or detector 8. Contaminated detector or injection port	1. Increase carrier gas source pressure to 15psig above column head pressure. 2. Check and correct as required (see page 4). 3. Replace septum with higher temperature type, and repack inlet side of column. Always use sharp syringe needle and inject in same place. Use of a needle guide is recommended. 4. Replace column with known good column. If okay, column requires reconditioning. 5. Measure flows and verify against manual specifications. 6. Allow time for instrument to equilibrate when changing operating temperature or installing another column. 7. Troubleshoot as in Isolation of Problem Source section. 8. Clean as recommended in instrument manual.
Symptom No. 8		
Irregular or unstable baseline Normal Problem	1. Column bleed or contamination 2. Contaminated detector or injection port 3. Carrier gas leak 4. Poor carrier gas regulation 5. Gas impurities or contaminated gas line 6. Gas flows not within minimum/maximum limits (including H_2 and air on FID) or poorly regulated flow 7. Defective electrometer, detector, or cable 8. FID only – collector incorrectly aligned 9. ECD only – heater wire too close to detector wire, causes AC noise	1. Check out with good column; if okay, recondition "bad" column. 2. Clean detector and/or injection port. 3. Check for and correct septum or column leaks (see page 4). 4. Check carrier gas supply for sufficient pressure. Replace tank if it is near empty. 5. Change gas tank, use gas purifier and clean metal tubing. 6. Measure flows and verify against manual specifications. 7. Troubleshoot as in Isolation of Problem Source section. 8. Realign as required. 9. Reposition heater wire.

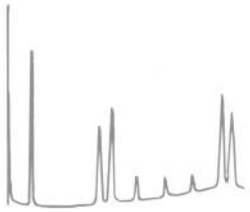
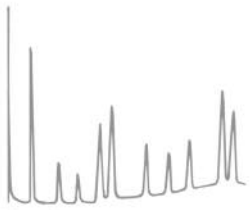
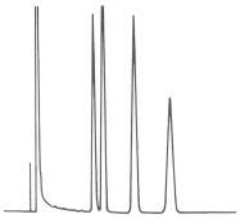
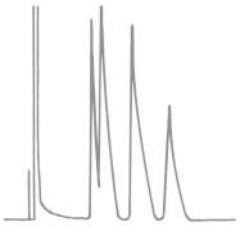
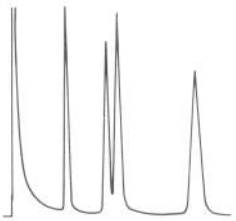
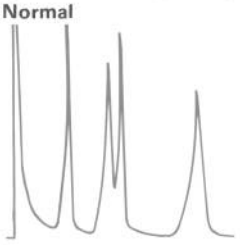
Symptom	Possible Cause	Remedy
Symptom No. 9		
Cycling baseline drift  	1. Poor instrument location (drafts, changes in ambient temperature, etc.) 2. Defective detector or oven temperature controller 3. Defective column oven temperature controller 4. Carrier gas flow irregular: insufficient supply pressure 5. Defective carrier gas regulator 6. Defective carrier gas flow controller 7. Sensitivity too high if using pumped gases, such as from hydrogen generator	1. Relocate instrument, close windows, etc. 2. Replace temperature sensing probe. 3. Replace temperature sensing probe. 4. Replace gas tank. 5. Replace regulator. 6. Replace flow controller. 7. Reduce detector sensitivity or decrease output pressure from generator.
Symptom No. 10		
Spikes (regular)  	1. Condensate or dust particles in FID 2. Contaminated gases 3. Defective electronics or detector	1. Clean detector and check ends of column to ensure glass wool is in place. 2. Replace gases or insert liquid nitrogen trap in gas line. 3. Troubleshoot electronics and detector as in Isolation of Problem Source section. Closely check the recorder cables and detector/electrometer cables.
Symptom No. 11		
Spikes (irregular or erratic)  	1. Defective cable, intermittent shorting 2. ECD heater wires and detector wire too close, or loose 3. FID – insufficient H_2 flow 4. Electronic interference from external source	1. Replace cable. 2. Check wire position and relocate if necessary. 3. Increase H_2 flow. 4. Relocate instrument, and determine possible interference sources (e.g. nearby transmitter site, etc.).
Symptom No. 12		
Negative Peaks  	1. Recorder improperly connected, polarity reversed, or sample injected into wrong column 2. TCD only – impurity in carrier gas	1. Reverse recorder connections or polarity switch. 2. Install or replace carrier gas purifier.

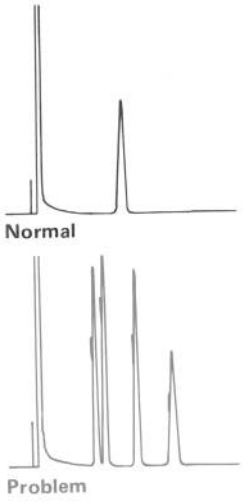
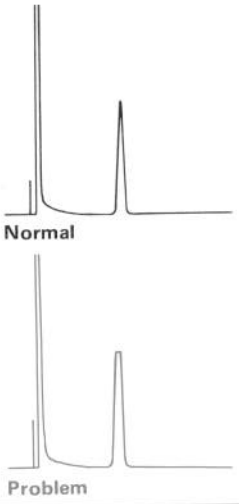
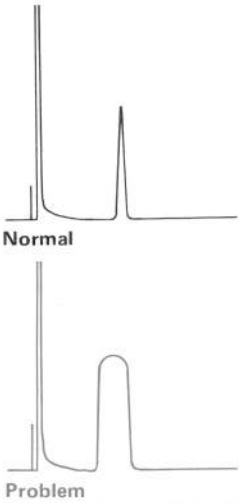
Symptom	Possible Cause	Remedy
Symptom No. 13		
Ghost peaks (peaks similar to previous sample appear when solvent alone is injected)  **Previous Sample**  **Normal** (solvent injected after sample)  **Problem** (solvent injected after sample)	1. Dirty syringe 2. Column adsorption and subsequent desorption of sample (particularly in temperature program) 3. Adsorption in transfer line	1. Try new syringe with clean solvent. If ghost peaks disappear, clean syringes more thoroughly. 2. Try more inert column materials (i.e. tubing, packing and glass wool). 3. Use glass-lined stainless steel for transfer lines.
Symptom No. 14		
"Extra" peaks (unlike sample chromatogram)  **Normal**  **Problem**	1. Septum bleed, particularly in temperature program 2. Peaks from previous runs, particularly if very broad with short retention time 3. Impurities from sample, solvent, sample container (e.g. plasticizer from cap liners or contaminated glassware), labware and reagents used in sample preparation, particularly when excess reagents are concentrated in work-up 4. Condensed carrier gas impurities eluting during temperature programming 5. Trace impurities in lab atmosphere	1. Turn off injector heater. If ghost peaks disappear, operate at lower injector temperatures or use high temperature septum. 2. Let analysis run longer and then repeat. 3. Run solvent blank with clean syringe. If extra peaks appear, change solvent; if no extra peaks appear, run solvent blank through entire sample work-up without sample. If no extra peaks appear, impurities are from sample. If extra peaks appear, repeat solvent blank from each step of work-up to isolate source. 4. Install or replace carrier gas purifier. 5. Verify by analyzing lab environment. Take corrective action as necessary.

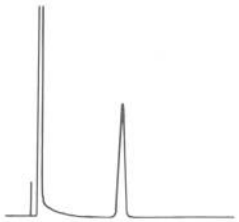
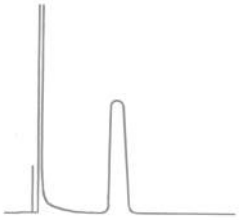
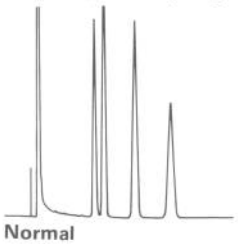
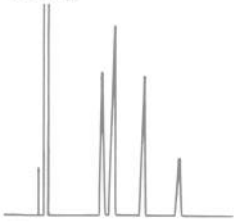
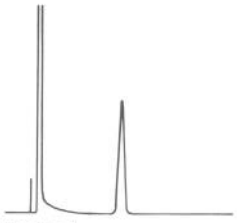
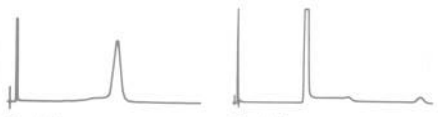
Column breakdown due to O_2 getting into system.

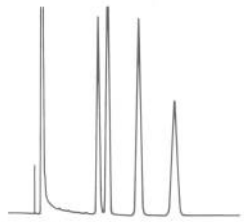
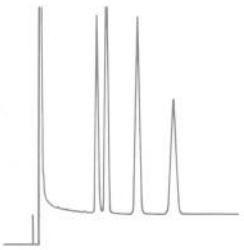
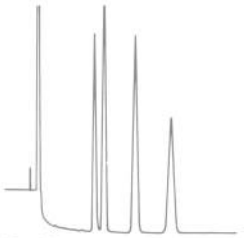
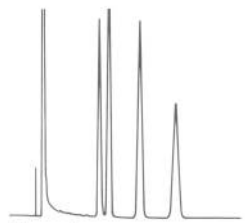
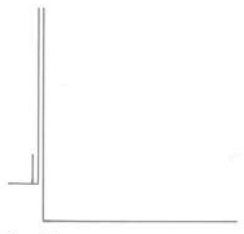
carrier gas contamination

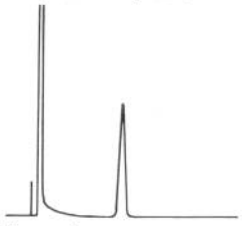
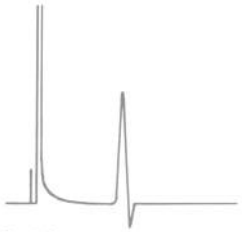
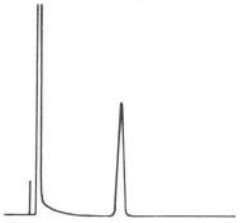
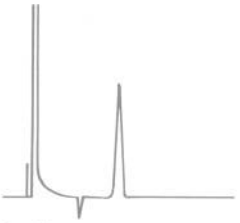
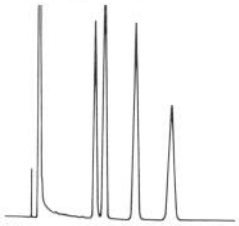
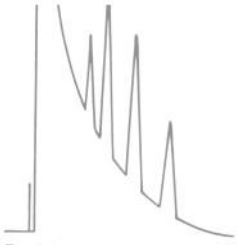
Fuel gases contamination

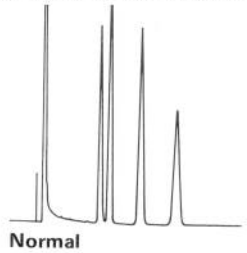
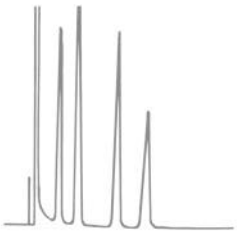
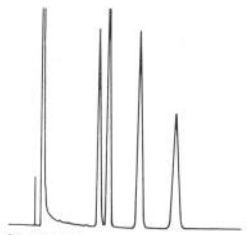
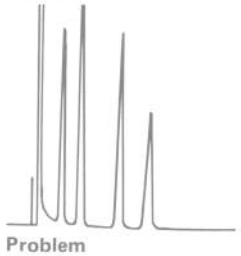
Symptom	Possible Cause	Remedy
(Symptom No. 14 cont'd.)  Problem  Problem	6. Air peaks or H_2O peaks 7. Multiple (or incomplete) derivatives formed in sample work-up 8. Sample decomposition	6. This is not a trouble symptom, but normal with TCD, using syringe injection or an aqueous sample. 7. Re-evaluate derivatization procedure. 8. Try lower temperature and/or different column.
Symptom No. 15		
Tailing Peaks  Normal  Problem	1. Column or injection port temperature too low 2. Column deteriorating 3. Active sample adsorbing on injection port, transfer lines, column, or glass wool 4. Two compounds eluting 5. Needle hitting and breaking packing in column inlet	1. Increase temperature (do not exceed maximum temperature for column). 2. If peaks tail on old column but retention times have not changed from what they were when column was new, replacing first 6" of packing or replacing the precolumn may help. If retention times have changed, column probably should be replaced. 3. Use more inert system: "all glass" or Teflon®, specially designed packing, on-column injection, and proper glass wool type. 4. Increase sensitivity and reduce sample size. Reduce temperature approximately 20°C and look for partial separation. 5. Remove some packing so needle won't penetrate it.
Symptom No. 16		
Leading Peaks  Normal  Problem	1. Column overload 2. Two components eluting 3. Sample condensation 4. Sample decomposition	1. Decrease sample size. 2. Decrease sample size, increase sensitivity, and decrease temperature by 20°C. Look for partial separation. 3. Check injection port and column temperatures, and increase if necessary. 4. Use inert system and deactivated packing.

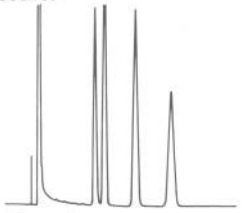
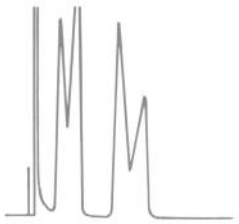
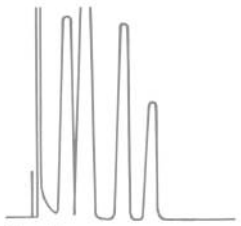
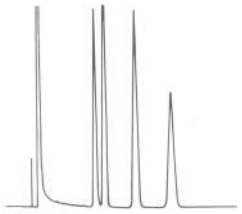
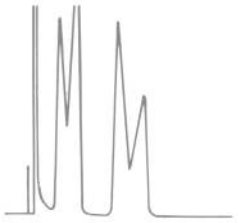
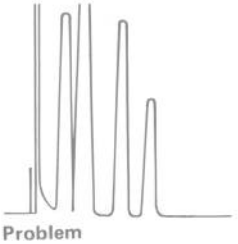
Symptom	Possible Cause	Remedy
Symptom No. 17		
Split Peaks 	1. Gross detector overload 2. Sample flashing prior to injection – simulates two injections	1. Reduce sample size. 2. Use syringe with solvent flush technique so that sample is contained in barrel, not in needle. Use less volatile solvent.
Symptom No. 18		
Square or flat top peaks 	1. Electrometer saturated (normal for solvent) 2. Defective recorder	1. Reduce sample size. 2. Troubleshoot recorder per recorder manual.
Symptom No. 19		
Round top peaks 	1. Detector overload – FID 2. Recorder gain too low	1. Decrease sample size. 2. Adjust gain control.

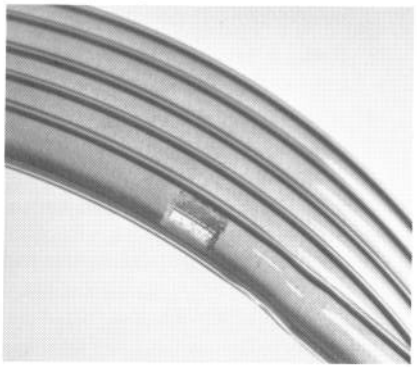
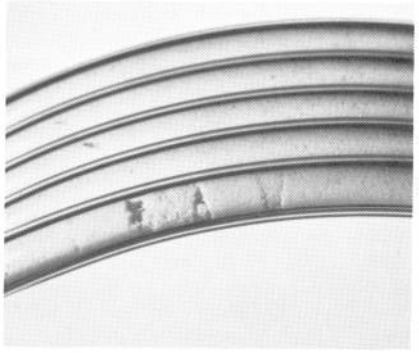
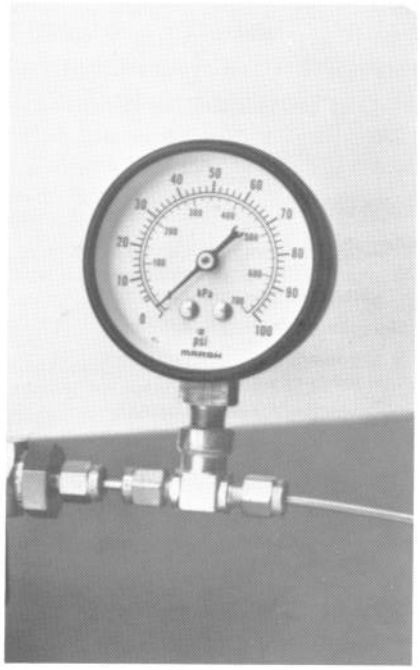
Symptom	Possible Cause	Remedy
Symptom No. 20		
Cigar top peak  Normal  Problem	Detector overload – ECD	Reduce sample size.
Symptom No. 21		
Clipped peaks – column efficiency exceptionally high  Normal  Problem	Recorder or instrument zero below minimum moveable range of recorder pen	Shunt recorder leads and set recorder baseline adjustment zero to approximately 5% of full scale.
Symptom No. 22		
Baseline rise before or after peak  Normal  Problem Problem	Sample decomposition	Use inert columns and packings.

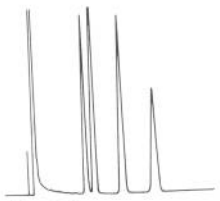
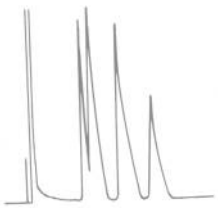
Symptom	Possible Cause	Remedy
Symptom No. 23		
Baseline change after large peak  Normal  Problem  Problem	1. Contamination – water or large component is stripping contaminants from column 2. Column not conditioned properly – liquid phase being stripped 3. Pressure imbalance when gas sampling valve activated	1. Repack first 6" of column or replace pre-column. 2. Recondition column. 3. Correct pressure imbalance.
Symptom No. 24		
Baseline drop after peak – FID only – flame extinguished  Normal  Problem	1. Sample too large 2. Incorrect gas flows 3. Flame tip plugged 4. Collector and tip not located properly: whistling or humming noise often heard	1. Decrease sample size. 2. Check and adjust carrier gas, H_2, and air. 3. Clean or replace flame tip. 4. Adjust collector position.

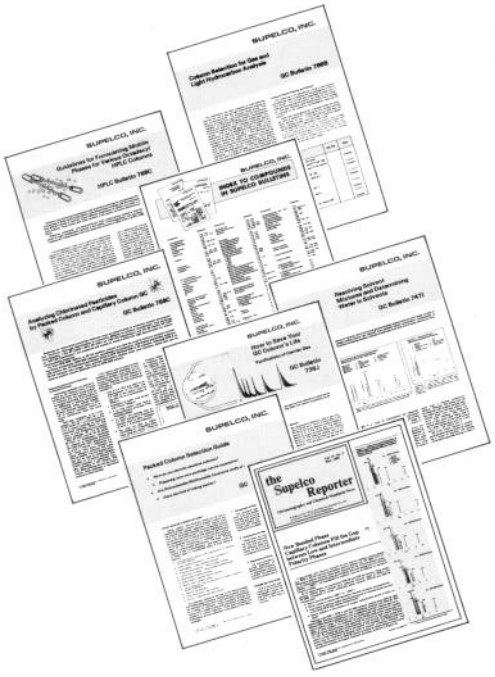
Symptom	Possible Cause	Remedy
Symptom No. 25		
Negative dips after peaks  Normal  Problem	1. Only after large peak such as solvent: sample too large 2. After all peaks with ECD: dirty detector cell	1. Decrease sample size. 2. Clean detector.
Symptom No. 26		
Negative dip before peak  Normal  Problem	1. Pressure imbalance when gas sampling valve activated	1. Correct pressure imbalance.
Symptom No. 27		
Broad solvent peaks  Normal  Problem	1. Dead volume in injection port due to poor column installation 2. Normal with very dilute sample as in trace analysis 3. Poor injection technique 4. Injection port temperature too low 5. Sample solvent interacts with the detector 6. Sample solvent retained by column (e.g. methanol by active columns)	1. Use on-column injection. Ensure proper column connections, particularly when changing from one column diameter to another. 2. – 3. Use smooth, rapid injection (see page 4). 4. Increase injection port temperature. 5. Change sample solvent. 6. Change sample solvent.

Symptom	Possible Cause	Remedy
Symptom No. 28		
Retention time longer (or shorter) on same column  Normal Problem  Problem	1. Column temperature too high (too low) 2. Carrier gas flow rate too low (too high) 3. Septum or column leak 4. Column contamination or deterioration 5. Recorder problem 6. Sample overload	1. Check temperature with independent thermometer, and adjust temperature as required. 2. Measure flow rate with soap bubble flow meter at column exit and adjust as required. 3. Check and correct as required. 4. Repack first 6" of column, or replace column. 5. Check recorder chart speed. 6. Reduce sample size.
Retention time on new column is longer (or shorter) than on previous column of the same composition  Normal Problem  Problem	1. Column too long (too short) 2. More (less) packing in column due to: a. Support density greater (less) than previously used b. Column packed more tightly (loosely) c. Column inside diameter greater (smaller) 3. Percent coating of stationary phase on support is too high (low) due either to different manufacturing procedures or errors 4. Different stationary phase used, or slight variation in phase composition (particularly commercial chemicals not manufactured for GC) 5. Different support type	This problem can be remedied by using pretested, standardized columns and packing materials.

Symptom	Possible Cause	Remedy
Symptom No. 29		
Unresolved peaks, on column which previously produced good results  Normal  Problem  Problem	1. Wrong column temperature 2. Wrong carrier gas flow rate 3. Sample problem a. Sample size too large b. Sample concentration different than previous analysis, causing "swamping" of minor peak by major peak 4. Poor injection techniques (slow) 5. Column contaminated or deteriorated	1. Check and adjust temperature. 2. Check and adjust flow rate. 3. a. Reduce sample size. b. Reduce sample size. 4. Use smooth, rapid injection. 5. Repack first 6" of column, or replace column.
Unresolved peaks, after previous column of the same composition produced good results  Normal  Problem  Problem	1. Column too long (too short) 2. More (less) packing in column due to: a. change in support density b. packed more tightly (loosely) c. column inner diameter different 3. Percent coating increased (decreased) either due to different manufacturing procedures or errors 4. Different stationary phase used, or slight variation in phase composition (particularly commercial chemicals not manufactured for GC) 5. Different support type	Each of these problem sources can be remedied by using pretested, standardized columns and packing materials.

Symptom	Possible Cause	Remedy
Symptom No. 30		
Large gaps appear in packing in glass column 	Improperly packed column	Add enough packing to fill in voids, then gently vibrate until smooth. If this does not solve the problem, column should be repacked.
Symptom No. 31		
Packing compacting or shrinking after conditioning 	1. Slight compacting may occur when packings are exposed to pressure 2. Large shift (3" or greater)	1. You can use this column. Shifts are not normally a problem. 2. Column may not be properly packed. Add more packing.
Symptom No. 32		
Low carrier gas flow and high pressure drop 	1. Overtightened septum 2. Insufficient carrier gas source pressure 3. Insufficient source pressure for temperature program 4. Plugged injection port, carrier gas line, or gas purifiers 5. Column over-packed or glass wool too tight	1. Loosen septum. 2. Increase gauge pressure by 10psig. 3. Flow control must have 10-15psig higher than maximum pressure (reached at maximum temperature) to function properly. 4. Replace tubing or gas purifiers as required. 5. Increase carrier gas pressure. (Note: not all packings have same pressure drop.) If flow is still insufficient, install another column. If flow is okay, column was the problem. If not, check plumbing system for flow restrictions (e.g. plugged detector, plugged gas filters, etc.).

Symptom	Possible Cause	Remedy
Symptom No. 33		
Column deteriorates too soon after installation (peaks tail, are poorly resolved, etc.)  Normal  Problem	1. Operating column near or above maximum temperature limit of packing 2. Water or O_2 in carrier gas is contaminating column 3. O_2 contamination due to column leaks 4. Aqueous samples, serum, plasma and other complex samples	1. a. Use higher temperature phase. b. Use shorter column and lower temperature if possible. c. Reduce temperature when column is not in use. d. Remove column when another column is used at higher temperature. 2. Use carrier gas purifier and appropriate grades of gases. Replace tanks before pressure becomes too low (300psig). 3. Check for leaks prior to use. Allow columns to cool before removing from GC to prevent column exposure to air while hot. 4. These samples can (1) strip phase from support, (2) chemically react with phase, (3) build up on column and possibly destroy the column, injection side first. Use of pre-column, or repacking the inlet will extend column life.



List of Bulletins by Subject
(Analyses are by GC, unless otherwise indicated)

FREE Technical Literature to Help You Select the Right Column for Your Analysis

Consistent and accurate chromatographic analyses are dependent not only on proper system operation, but also on packing materials and analysis techniques.

Examine the following list of Supelco Bulletins. Select the right one (or more) to help you perform your particular analysis.

	Bulletin Number
ACIDS (ORGANIC)	
Dibasic (Dicarboxylic) Acid Methyl Esters (up to C10)	762
Elaidate/Oleate — separation of cis/trans isomers	752
Fatty Acids, Free (C14-C20)	727
Fatty Acid Methyl Esters (C14-C24)	746
Fatty Acid Methyl Esters (postional cis/trans isomers)	822
Fatty Acids, Volatile (C2-C5) — at ppm levels in water	751
Fatty Acids, Volatile and Nonvolatile — esterification and separation	721
ALCOHOLS (also see SOLVENTS)	
Acylation	721
Alcoholic Beverage Analyses	790
Blood Alcohols **(GC/HPLC)**	810

	Bulletin Number
AMINES	
Acylation	721
Aliphatic and Aromatic Amines — acylation, columns, separations	737
Nitrosamine Priority Pollutants	775
N-Nitrosamines — contaminants in foods or wastewater	791
Phenylenediamines **(GC/HPLC)**	805
AMINO ACIDS, PROTEINS	
Homologs and Polymers — separation by size exclusion HPLC **(HPLC)**	796
Proteins **(HPLC)**	795
APPARATUS	
Capillary Column Butt Connector	801
Glass GC Columns of Consistent Dimensions	804